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Shaughnessy No.: 105501

Date Out of EAB: NOV 7 1988

To: Robert Taylor
Product Manager #25
Registration Division (TS 767)

From: Paul Mastradone, Acting Chief
Environmental Chemistry Review Section #1
Environmental Fate and Ground Water Branch

Thru: Henry Jacoby, Acting Chief
Environmental Fate and Ground Water Branch

Henry Jacoby

Attached, please find the EAB review of:

Reg./File Symbol: 1471-101

Chemical Name: Tebuthiuron

Type Product: Herbicide

Product Name: SPIKE, Graslan

Company Name: Elanco(Div. of Eli Lilly)

Purpose: Review two leaching studies

Date Received: 08/17/88

Action Code: 660

Date Completed: 10/06/88

EAB #(s): 80929

Monitoring Study Requested: _____

Total Reviewing Time: 4.0 days

Monitoring Study Voluntarily: _____

Deferrals To:

_____ Ecological Effects Branch

_____ Dietary Exposure Branch

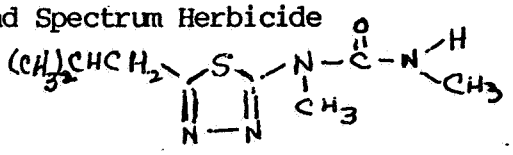
_____ Toxicology Branch

1. Chemical: Tebuthiuron

Trade Name: SPIKE, Graslan

Chemical Name: N-[5-(1,1-dimethylethyl)-1,3,4-thiadiazol-2-yl]-N,N'-dimethylurea

Type of Pescitide: Broad Spectrum Herbicide

Structure: 

2. Test Material: see DER

3. Study/Action Type: Review adsorption/desorption and aged column leaching studies

4. Study Identification:

Mobility of Tebuthiuron in Soil. D. G. Saunders and F. L. Powers. Performed by Lilly Research Lab, Greenfield, Ind. Submitted by Elanco Products Company (a division of Eli Lilly and Co.) July 29, 1988. Accession # 407684-01.

5. Reviewed By:

Patricia Ott
Chemist
Environmental Chemistry Review Section #1

Signature: Patricia Ott
Date: 10/27/88

6. Approved By:

Paul Mastradone
Acting Chief
Environmental Chemistry Review Section #1

Signature: Paul J Mastradone
Date: NOV 7 1988

7. Conclusions:

a. Adsorption/Desorption (unaged)

This study satisfies the Subpart N Environmental Chemistry Guidelines requirement for unaged leaching and shows that tebuthiuron is mobile in sand, sandy loam, loam, and clay loam with K_a's of 0.11, 0.62, 0.82, and 1.82, respectively.

b. Aged Column Leaching

This study satisfies the Subpart N Environmental Chemistry Guidelines data requirement for aged leaching (the soil column was inverted and the leachate fractions were collected at the top). This technique is acceptable, based on a conversation with Dr. Charles S. Helling. The sandy loam soil was aged for 30 days, and 97% of the radioactivity was present as parent, so no further work is needed at this time. About 1-2% of the applied radioactivity was metabolite 109383 (N-[5-(1,1-dimethylethyl)-1,3,4-thiadiazole2-yl]-N-metyhlurea), which appears to be mobile, also. If there is a concern about any degradate(s) in the future, a leaching study may be required for the degradate(s) of interest.

The information in this study indicates that tebuthiuron can move through the

soil profile.

8. Recommendations:

The unaged leaching data requirement is satisifed and no further work is recommended for the aged leaching requirement, at this time. However, if there is a concern in the future about any degradate(s), then a leaching study may be required on the degradate(s) of interest.

9. Background:

This data was submitted in resonse to the Registration Standard (July 1987). Tebuthiuron is on the GWDCI list and has been found in shallow Texas ground water, and is on the list of analytes to be monitored in the National Pesticides in Ground Water Monitoring Survey. The Ground Water Team has recommended a small-scale retrospective study.

10. Review of Individual Studies: see DER

11. Completion of One-Liner: attached

12. CBI Appendix: N/A

DATA EVALUATION RECORD

Tebuthiuron Study 1 (Unaged Adsorption/Desorption and Aged Soil Column)

Mobility of Tebuthiuron in Soil. D. G. Saunders and F. L. Powers. Performed by Lilly Research Lab, Greensfield, Ind. Submitted by Elanco Products Company (a division of Eli Lilly and Co.). July 29, 1988. Accession # 407684-01.

Reviewed By: Patricia Ott Signature: Pat Ott
Title: Chemist Date: 10/27/88
Org: Environmental Chemistry Review Section #1

Approved By: Paul Mastradone Signature: Paul J Mastradone
Title: Acting Chief Date: NOV 7 1988
Org: Environmental Chemistry Review Section #1

Conclusions:

1. Adsorption/Desorption Experiment (4 unaged soils)

The adsorption/desorption study satisfies the Subpart N Environmental Chemistry Guidelines data requirement for unaged leaching by providing a quantitative estimate of mobility of tebuthiuron in 4 soils (sand, sandy loam, loam, and clay loam).

Tebuthiuron is mobile in all 4 soils and has a K_a for sand, sandy loam, loam, and clay loam of 0.11, 0.62, 0.82, and 1.82, respectively.

2. Aged Sandy Loam Soil Column Experiment

This study satisifies the Subpart N Environmental Chemistry Guidelines data requirement for an aged leaching study (the soil column was inverted and leachate fractions were collected at the top). The data shows that ^{14}Ctebuthiuron aged for 30 days in sandy loam soil, then topped with untreated sand or loam, can move through the soil profile.

Tebuthiuron persists in soil and accounted for 97% of the applied radioactivity in the sandy loam soil aged 30 days (which was used in the aged soil column study). Additional leaching studies for tebuthiuron degradates may be required in the future, depending on the results of the studies which will be submitted to fill other environmental fate data gaps.

Metabolite 109383 (N-[5-(1,1-dimethylethyl)-1,3,4-thiadiazole-2-yl]-N-methyl-urea) was present at 1-2% of the applied radioactivity, corrected for the amount present in the radiolabelled tebuthiuron standard (1.4%).

Materials and Methods:

Two experiments were done: adsorption/desorption using unaged sand, sandy loam, loam, and clay loam) and an aged soil column leaching study using sandy loam. Radiolabelled ^{14}C-tebuthiuron (5-thiadiazole ring labelled and 98% pure) was used.

The ^{14}C material contained 1.4% of compound 109383 (N-[5-(1,1-dimethylethyl)-1,3,4-thiadiazole-2-yl]-N-methylurea). The standard reference compound for this degradate had a purity of 99%. (Soil characteristics are attached.)

Soils were taken from Texas and Indiana, sampled from the "A" horizon and were air dried, crushed gently, sieved, and stored frozen until used. Soil adsorption/desorption results were calculated on a dry soil weight basis.

1. Adsorption/Desorption Experiment

Adsorption studies were conducted in 50 ml glass centrifuge tubes. Twenty grams of soil was added plus 40 ml of one of 4 different ^{14}C-tebuthiuron solutions (5, 1, 0.2, and 0.04 ug/ml, prepared using 0.01 M calcium chloride). The tubes were mixed and equilibrated at 25°C, then centrifuged. A 2 ml aliquot was taken and ^{14}C content determined by LSC.

The appropriate equilibration period of 22 hours was previously determined by an adsorption time study using sandy loam and clay loam. For desorption, 20 ml of the aqueous layer was discarded and replaced with 22 ml of 0.01 M calcium chloride. The soil was resuspended and mixed for 22 hours. After centrifugation, 2 ml aliquots were counted by LSC. A second desorption was also completed.

An additional experiment was conducted to determine the extent of adsorption of ^{14}C-tebuthiuron on the inside of the glass centrifuge tube. Thirteen tubes containing no soil were prepared as described above and mixed for 22 hours. A 2 ml aliquot was counted for each tube.

2. Aged Soil Column Leaching (Reverse Column)

^{14}C-Tebuthiuron treated sandy loam soil, fortified with the equivalent of 6 lb/acre (maximum application rate) was aged in a glass bottle connected to an air flow and trapping system (charcoal trap for organics and CO_2 trap). See Figure 1.

The leaching study was conducted with the soil columns (3.6 cm i.d. x 30 cm long Teflon tubing) inverted. Glass wool was put on the bottom, then 0.5 cm of untreated sandy loam, 3 cm treated sandy loam, and on top was 26.5 cm of untreated sand or loam. Leachate fractions were collected at the top. Columns were maintained at 25°C. See Figures 2 and 3.

Even though leaching in this study actually occurred with the leachate flowing upward, it is discussed in the report as if the leachate passed down through the column. Soil depths are also measured from the surface down.

After columns were saturated with 0.01 M $CaCl_2$, leaching solution (50.8 cm or 520 ml, which is the correct amount) was added and collected in 4 increments from Feb. 15, 1988 to Feb. 19, 1988. Then the columns were inverted and allowed to drain for 2 days. The soil was extruded and divided into five 6 cm segments. Subsamples were combusted to determine ^{14}C content.

Soil was extracted by refluxing 3 hours with a 1:4 aqueous methanol solution. The extract was separated from the soil, which was washed with water followed by methanol. After drying, the extracted soil was ground, combusted and analyzed by LSC.

The extract and 2 washes were combined and counted. A portion was removed and aqueous sodium chloride added. The radioactivity was extracted 3x with dichloromethane and ^{14}C content determined on an aliquot by LSC. The remaining extract was concentrated and analyzed by TLC.

The leachate was extracted 3x with dichloromethane and a portion analyzed by LSC. The aqueous layer was extracted with ethyl acetate and analyzed by LSC. The extracts were analyzed by TLC, also.

The charcoal in the charcoal tube was extracted twice for 30 mins each using methanol, and the extract was analyzed by LSC.

Extracted soil and charcoal was combusted and the $^{14}CO_2$ analyzed by LSC.

TLC silica gel plates were developed using 90:10:1 chloroform/methanol/ammonium hydroxide. Radioactive zones were detected by radioautography, and the areas of interest were scraped off, extracted with a 4:1 methanol/acetic acid solution, then analyzed by LSC.

A storage stability test was done by fortifying soil with ^{14}C-tebuthiuron and holding the smaples at -20°C for about 2 months, then analyzed as described above. No degradation occurred during storage.

II. Reported Results:

1. Adsorption/Desorption

Soil-water adsorption coefficients (K_a's) found were:

Soil	K_a	%OM
Sand	0.11	0.5
Sandy Loam	0.62	1.4
Loam	0.82	1.8
Clay Loam	1.82	2.0

The greater the amount of organic matter in the soil, the greater degree of adsorption of tebuthiuron occurred.

2. Aged Soil Column Leaching

A summary of the material balance at the end of the 30 day aging period for sandy loam is given below:

Fraction	% of Initial
Aged soil	98.38
Charcoal tube extract	0.007
Charcoal tube combustion	none detected
CO_2 trap	0.13
total:	98.51

TLC analysis of the aged sandy loam showed it contained 97% parent and 1 to 2% of metabolite 109383 (N-[5-(1,1-dimethylethyl)-1,3,4-thiadiazole-2-yl]-N-methyl-urea), corrected for the 1.4% present in the ^{14}C-tebuthiuron used to fortify the soil before aging.

The report said that the amounts of tebuthiuron and metabolite 109383 in the leachate are similar to the amounts present in the aged soil, suggesting that the metabolite is about as mobile as tebuthiuron, but no data was given to show this.

TABLE I

INFORMATION ON SOILS USED IN THE ADSORPTION STUDY

Soil No.	Textural Classification	Soil Series	Location	Date Collected	Sieve Mesh Size
1	Sand	Nueces	Hidalgo Co., TX	7/85	20
2	Sandy Loam	Fox	Johnson Co., IN	8/4/87	10
3	Loam	Crosby	Hancock Co., IN	8/4/87	10
4	Clay Loam	Brookston	Hancock Co., IN	8/4/87	10

TABLE II

CHARACTERISTICS OF SOILS USED IN THE ADSORPTION STUDY

Soil No.	Percent				pH	Cation Exchange Capacity meq/100 g	Bulk Density g/cc	Percent	
	Sand	Silt	Clay	Organic Matter				Loss on Drying	Moisture at Field Capacity
1	89.2	5.6	5.2	0.5	7.7	3.5	1.48	0.36	4.87
2	66	22	12	1.4	5.7	4.9	1.28	2.48	11.86
3	28	48	24	1.8	6.5	10.5	1.11	5.28	20.36
4	24	44	32	2.0	6.9	21.2	1.15	6.48	25.96

Tebuthiuron

Page_____ is not included in this copy.

Pages 9 through 13 are not included.

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____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

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